Willkommen
Welcome
Bienvenue

Material Aspects in Metal Additive Manufacturing

Challenges, Opportunities, Visions

Dr. Christian Leinenbach

Empa - Swiss Federal Laboratories for Materials Science and Technology

Outline

- Introduction – Current state of additive manufacturing of metals and alloys
- Challenges – new materials for additive manufacturing
- Opportunities – new materials by additive manufacturing
- Visions – components from new materials with new functionalities

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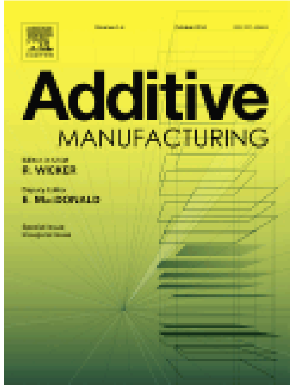
Materials aspects in AM - overview

- Increasing interest in material science aspects of AM
- MS&T 2014, Pittsburgh
  - Special session «Materials science of AM»
  - 51 contributions
- TMS Annual Meeting 2015, Orlando
  - Main symposium «Additive Manufacturing»
  - 77 contributions
- MS&T 2015, Columbus
  - Main symposium «Additive Manufacturing»
  - 4 sessions, «Additive Manufacturing of Metals», «In-situ Process Monitoring, Defect Dectection and Control», «Materials Science of Additive Manufacturing», «Novel Material and Process Development for Additive Manufacturing»
  - >140 contributions
- Euromat 2015, Warzaw
  - Session: Materials Processing – Additive Manufacturing

Materials aspects in AM - overview

- Journal of Materials Research, special issue September 2014 «Materials Science of additive manufacturing»
  - 34 papers on AM of metals, ceramics and polymers
- New Elsevier-journal «Additive Manufacturing»
  - "The journal covers a wide scope, comprising new technologies, processes, methods, **materials, systems,** and applications in the field of additive manufacturing"



The most widely used materials

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- ~190 contributions to the previously mentioned conferences and journals were on metal AM (status: February 2015)
  - ~38% Ti-6Al-4V (cp-Ti)
  - ~21% Inconel 718/625
  - ~17% Stainless Steel (316L, 304)
  - ~9% Al-alloys (AlMgSi, AlCu)
  - ~8% Intermetallics (NiTi, γ-TiAl)
  - ~5% CoCrMo alloys
  - ~2% others (Mg-alloys, noble metals, composites, HEA)

- The typical title is «Microstructure and mechanical properties of Ti6Al4V/In718/SS316L produced by SLM/EBM/DMD»

Materials of interest for AM

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- In Switzerland, there is a specific need for AM of the following materials
  - Advanced high-temperature alloys (γ'-hardening Ni-based alloys, Co-based alloys, TiAl) for power generation and aerospace applications
  - Tool steels, HSS, metal-superabrasives composites for advanced shape forming tools (grinding, cutting, milling etc.)
  - Precious metal alloys (Au-, Pd-, Pt-based) for jewelry and watches
  - Shape memory alloys (NiTi) for medical applications and micro-actuators

→ Useful information on the processability of those materials is very limited or not existing!

Parameters influencing the material properties

- The quality and the properties of AM manufactured components are strongly dependend on
  - AM processing technology (powder bed, powder feed, wire feed)
  - Energy transfer (laser, e-beam)
  - Beam shape
  - AM processing conditions (shielding gas, vacuum)
  - Scanning strategy – machine type
  - Scanning parameters (P_{las}, v_{las}, hatch distance, layer thickness)
  - Alloy powder shape, grain size & size distribution
  - Powder impurities
  - Pre-heating
  - …

→ The correlation between the different parameters and the material properties needs to be better understood!

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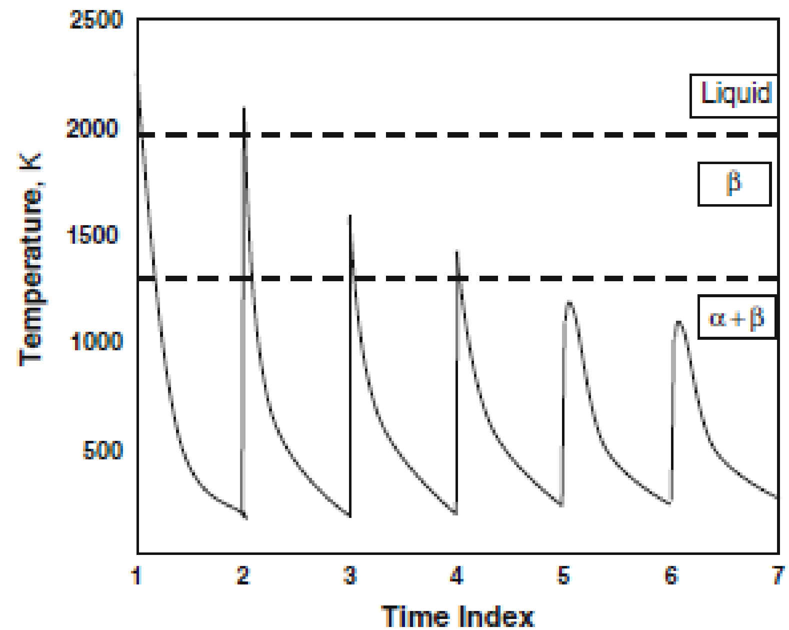
What are the problems with AM processing of technical relevant alloys?

- Fast heating and cooling ($\Delta T \approx 10^3$–10^5 K/s)
  - → suppressed phase transformations; supersaturated phases
  - → segregation
  - → hot cracking
  - → thermal residual stresses
- Unidirectional heat flow into building plate/substrate
  - → textured grains; anisotropic properties
- Every layer undergoes repeated heating and cooling cycles; temperatures can exceed T_{liq} or $T_{\alpha\leftrightarrow\beta}$
  - → Multiple phase transformations and complex microstructures; thermal residual stresses

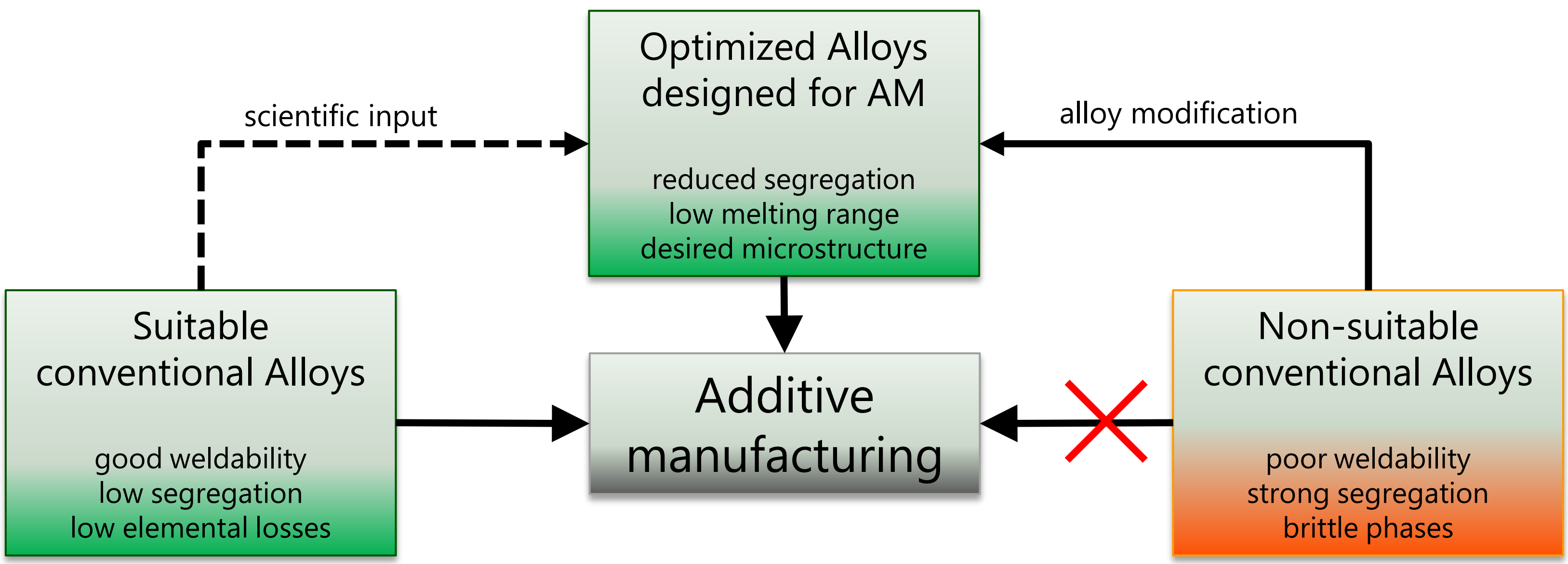
thermal profile of a single layer AM processed Ti-6Al-4V



/W.E. Frazier, J. Mater. Eng. Perform. 23 (2014) 1917/

What are the problems with AM processing of technical relevant alloys?

- The phase transformations in multi-component alloys under AM conditions (=rapid solidification) must be understood and controlled!
  - ➢ knowledge on stable and meta-stable phase diagrams required
  - ➢ knowledge on thermodynamic and thermophysical quantities required
  - ➢ knowledge on diffusion kinetics, mobilities required



Alloy development for AM – Empa approach

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- Ultimate test: AM using an optimized alloy
  - AM equipment
  - new alloy according to specifications
  - suitable powder shape

processability=f(process, powder, alloy)

- Intermediate test: Alloy behavior during rapid melting and cooling using the AM equipment
  - equipment for rapid heating and cooling (=AM equipment)
  - new alloy in solid form
  - no powder needed

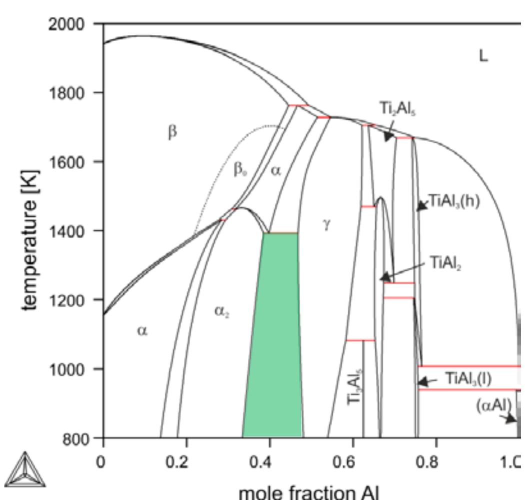
«processability»=f(process, alloy)

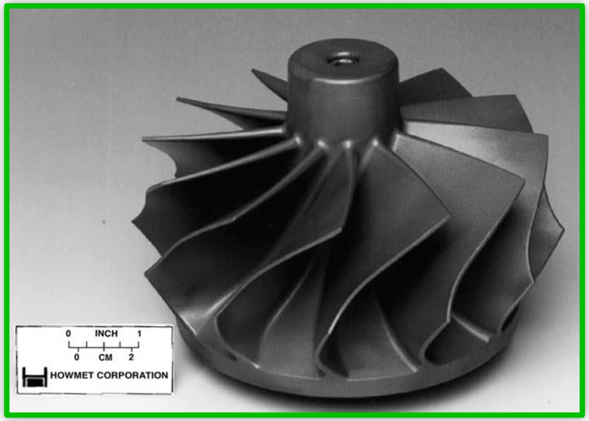
- First level test: Alloy behavior at high cooling rates
  - rapid cooling equipment (≠ AM equipment)
  - new alloy
  - no powder needed

«processability»=f(alloy)

Alloy development for AM – TiAl

- Ti-Al alloys of interest for high temperature structural components
  - low density (~3.9-4.2 g/cm^3)
  - high Young's modulus (~140 GPa), high strength, creep resistant
  - higher oxidation resistance than Ti alloys
  - higher service T than Ti alloys
- Fully intermetallic
  - low elongation to fracture, brittle at room temperature
  - sensitive to contamination, properties strongly dependent on phase morphology
  - Extremely difficult to process by AM





Rapid solidification – basic offline tests

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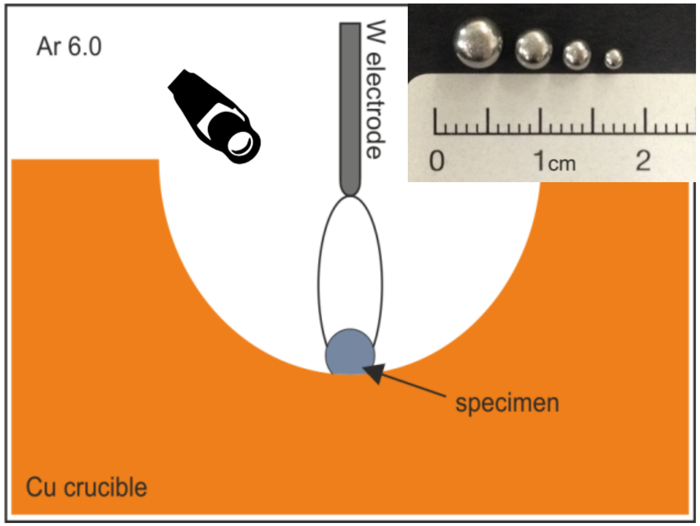
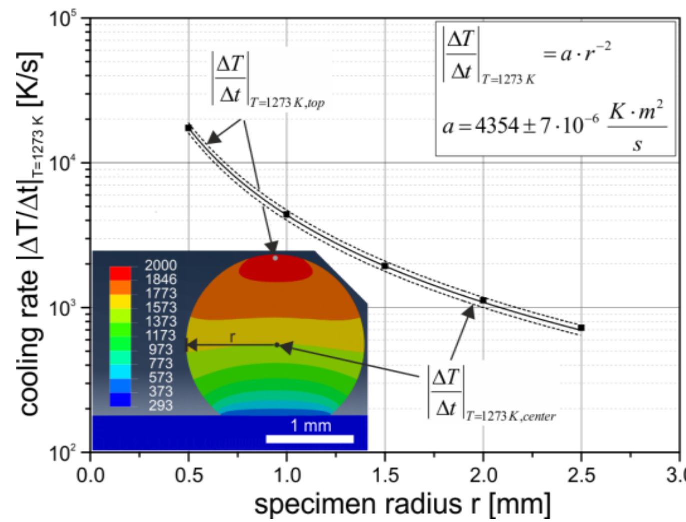
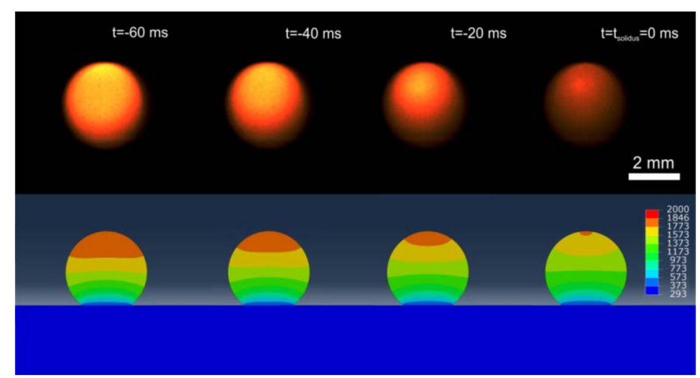


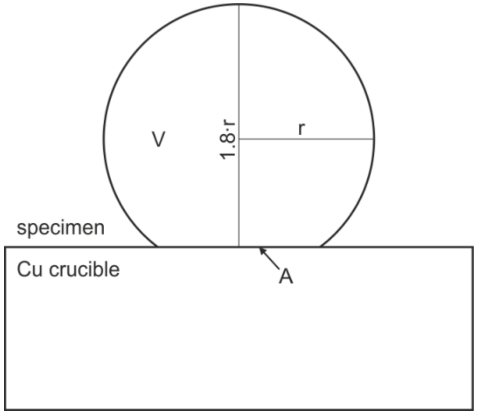


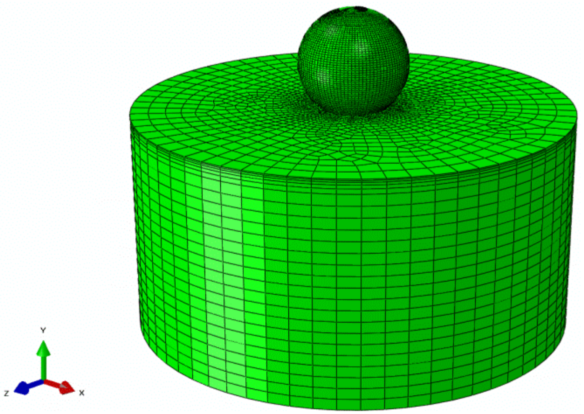
- heating and rapid solidification of small samples using W-electrode arc melting or laser beam melting
- size dependent cooling rates
  - spherical samples, the smaller the faster
  - cooling rate ~ r^{-2}
- function correlating radius and cooling rate
  - single «material» parameter to describe the complete curve
- simulation verification by high speed camera measurement
  - comparable solidus propagation in experiment and simulation

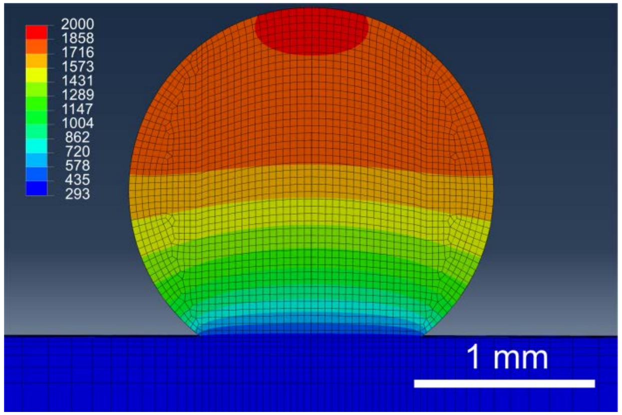


/Kenel C, Leinenbach C. J Alloys Compd 2015;637:242/

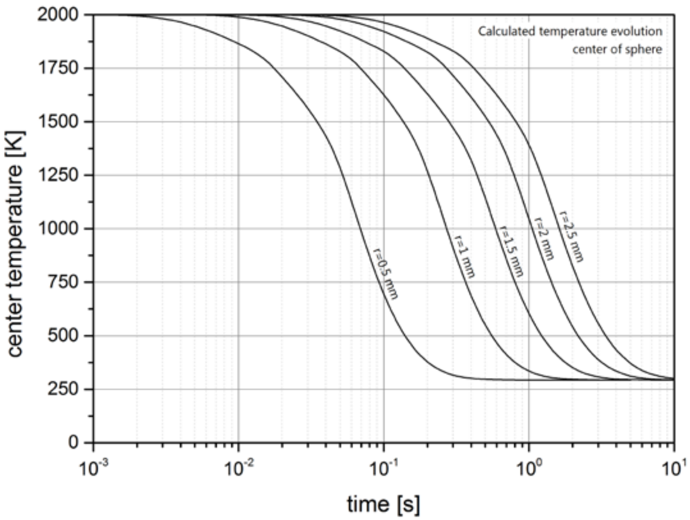
Rapid solidification – FE modeling







- hexahedral meshed part ~ 160’000 elements
  - 50 elements across sphere
  - dense mesh below sphere for accurate heat transport
- boundary conditions for cooled Cu part
  - side and lower surface T=293 K
- modelled heat flows
  - conductive transport sphere-substrate
  - radiation of surface to ambient surrounding
- phase transformations
  - enthalpy of fusion included for solidification



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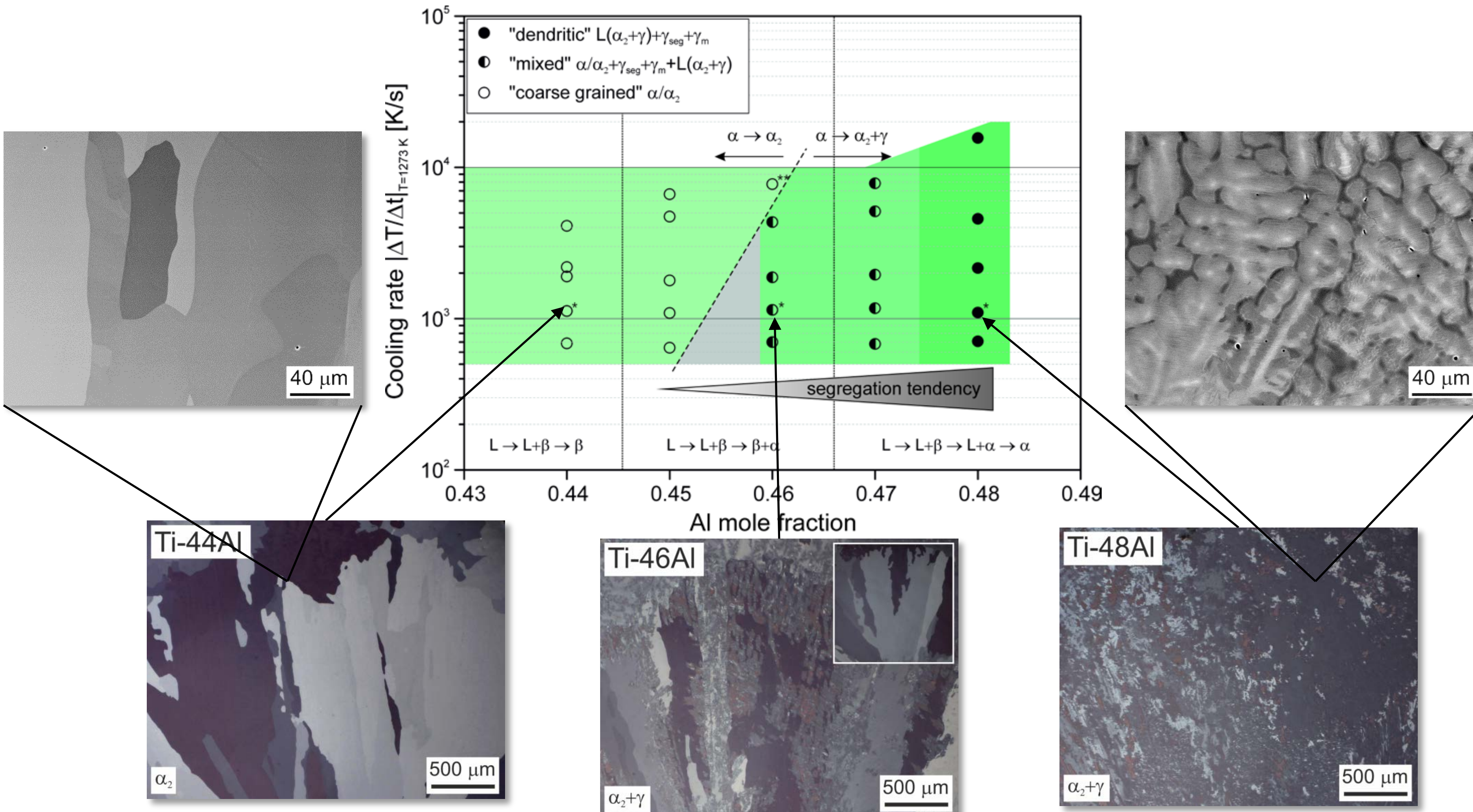
Influence of cooling rate on microstructure formation



Kenel C, Leinenbach C. J Alloys Compd 2015;637:242.

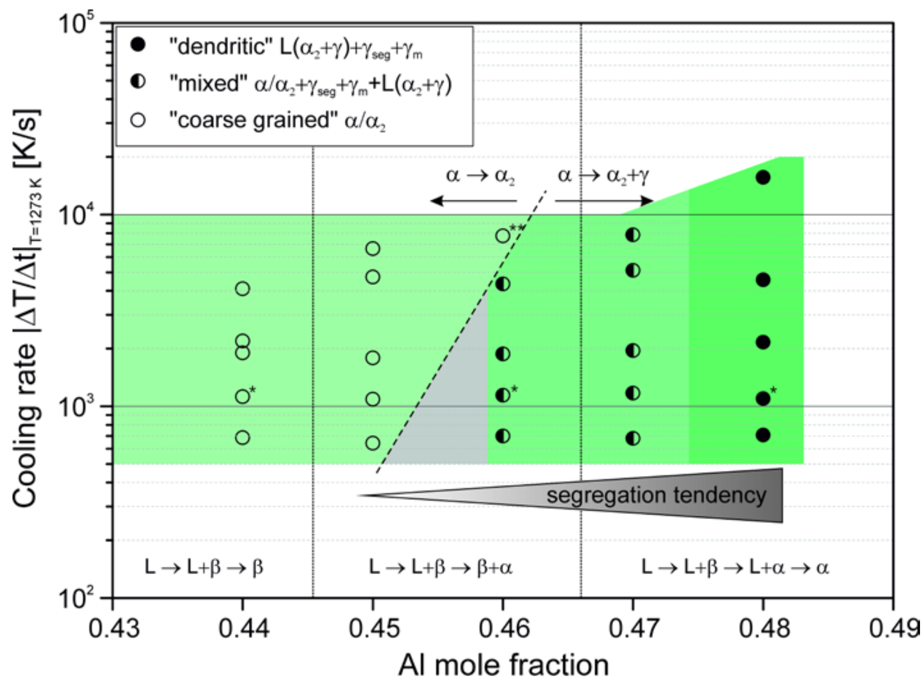
Influence of cooling rate on microstructure formation

$\alpha \rightarrow \alpha_2$ ordering

single phase

oversaturated α_2

extremely brittle



$\alpha \rightarrow \alpha_2+\gamma$

out-of-process two phase

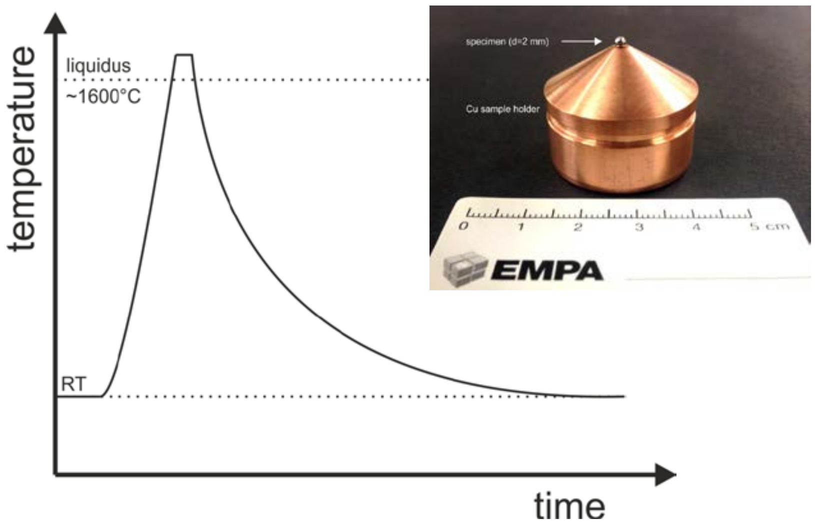
desired set of phases

tough

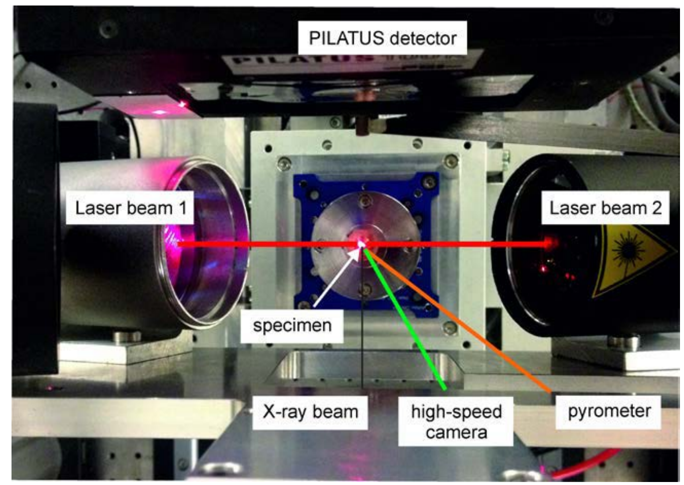
- composition – cooling rate – microstructure maps
  - properties relevant to processing (here: formation of intermetallic phases)
  - data for alloy selection
  - similar to processing window determination experiments → indications for suitable processing parameters
- predictability based on equilibrium phase diagram information: limited

In situ Synchrotron XRD on rapidly heated and cooled alloys

(with J. Fife, H. Van Swygenhoven, D. Grolimund, S. Van Petegem – Paul-Scherrer-Institute, Villigen, CH)

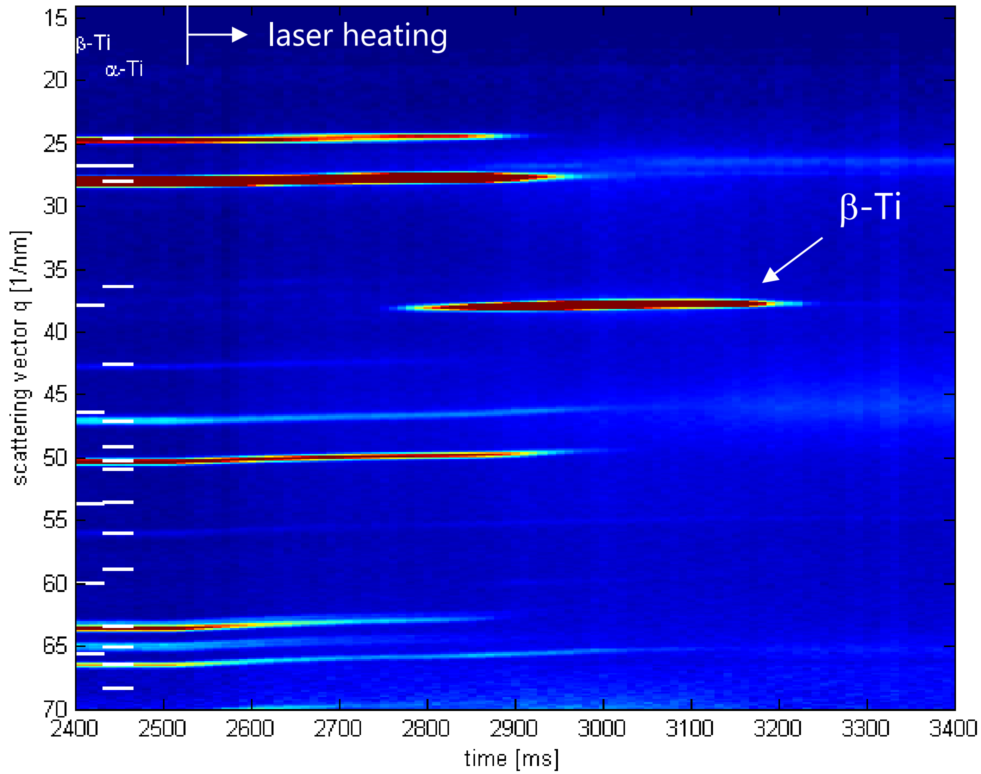


experimental setup, top view



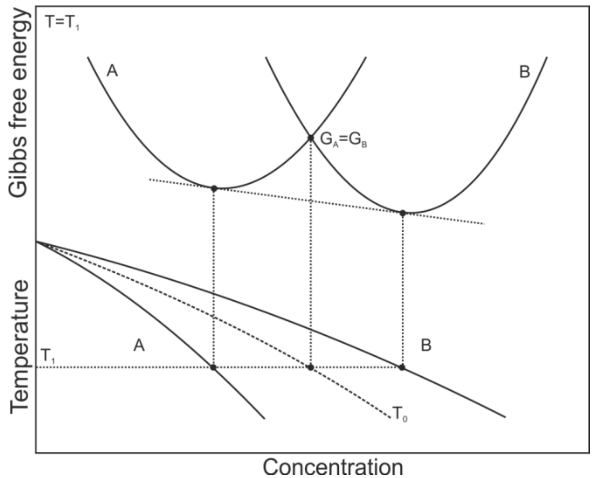
- setup of laser beam heating stage inside Synchrotron beam line at PSI
- *in situ* XRD during laser melting and soldification of Ti alloys
- feasability for controlled Ti- and TiAl melting and solidification
- high speed camera measurements for additional information

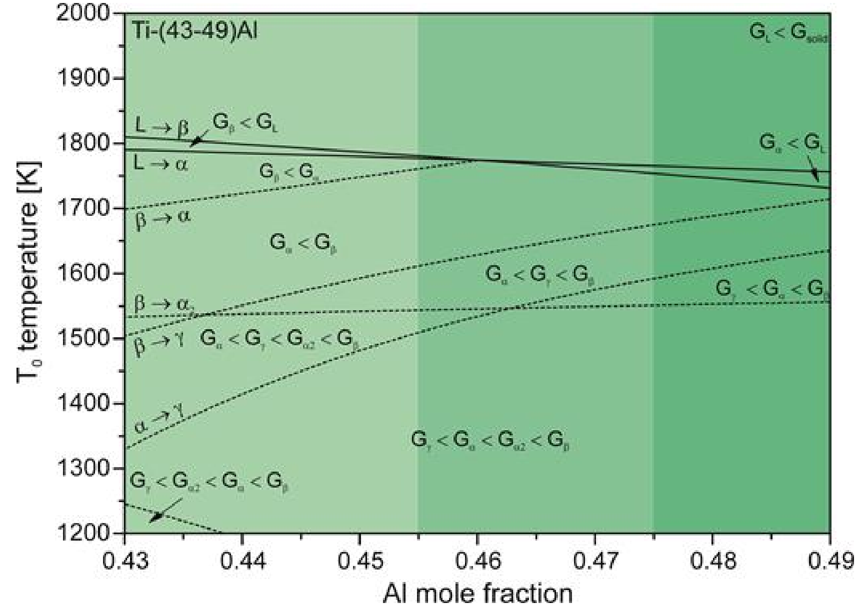
In situ XRD on rapidly heated and cooled alloys – preliminary results



- *in situ* (Laue) XRD during laser melting and solidification of Ti
- $\alpha \rightarrow \beta$ phase transformation and melting clearly observable
- high temporal resolution can be reached using synchrotron radiation
- facility can be used in other beamlines (e.g. tomography)

Development of phase selection hierarchy maps





- diffusion-less phase transformation
  - ideally no diffusion → all phases have the same composition
  - phase B transforms to A if $G_A<G_B$
- T_0 temperatures for different phase transformations and solidification
  - calculated using CALPHAD
  - based on published thermodynamic assessment for Ti-Al [1]
- map constituents
  - T_0 temperature curves for specific phase transformations
  - fields with a hierarchy according to the Gibb's free energy
- «phase diagram without diffusion»

/Kenel C, Leinenbach C. J Alloys Compd 2015;637:242/
/Witusiewicz VT et al. J Alloys Compd 2008;465:64./

Prediction of transformation behavior

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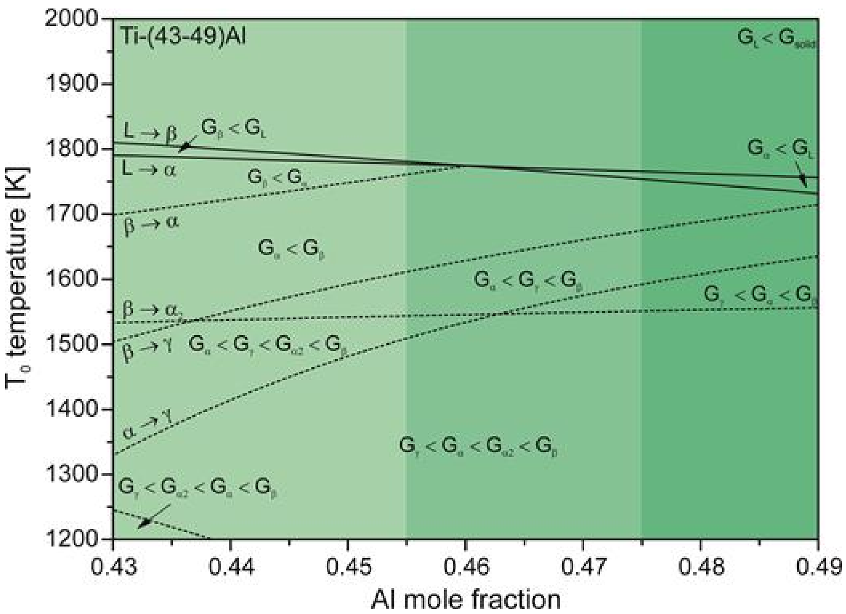
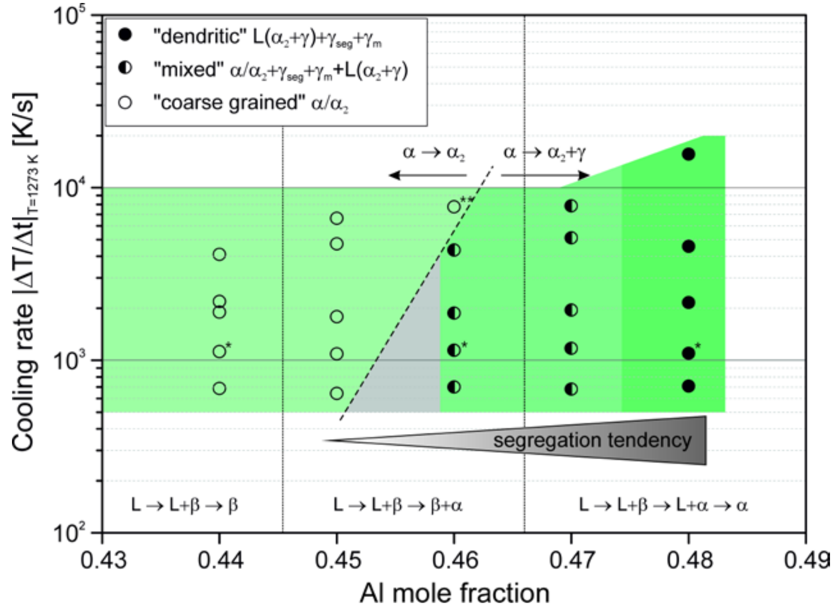
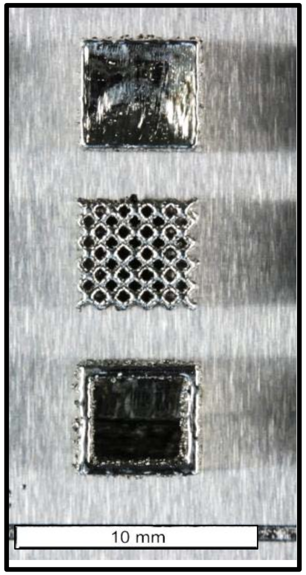
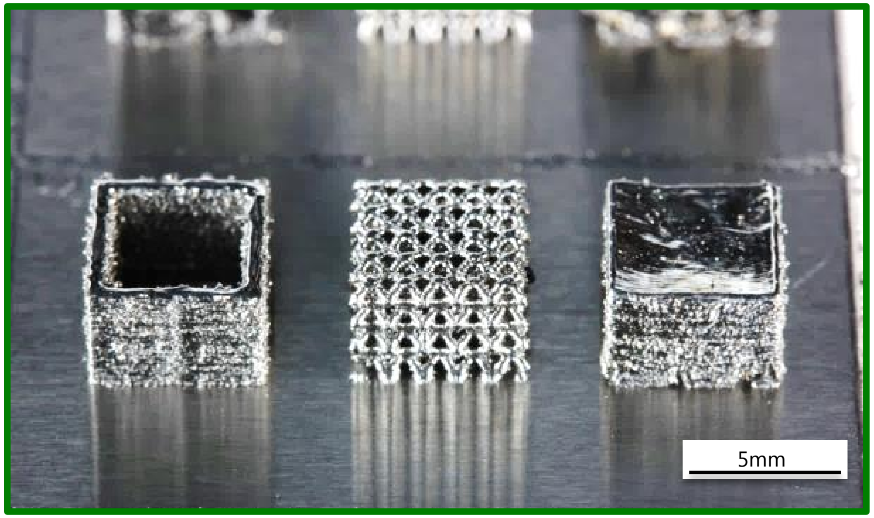




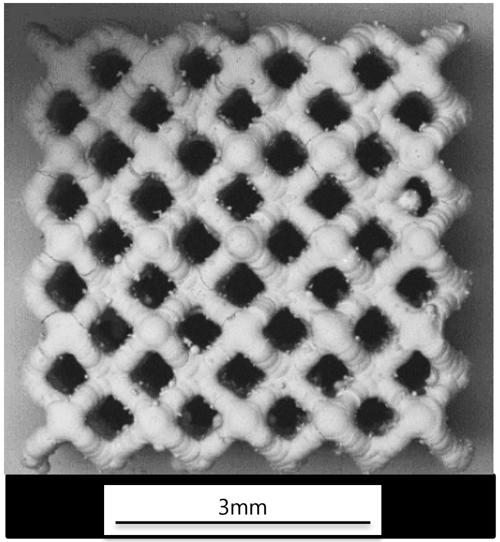
- diffusion-less T_0 concept allows to predict the changed solidification behavior
- deviation from equilibrium phase diagram can be explained
- influence of kinetics for Ti-46Al at studied cooling rate
- complementary tool for alloy development
  - pre-screening of alloys
  - understanding of experimental results
  - reduction of experimental effort

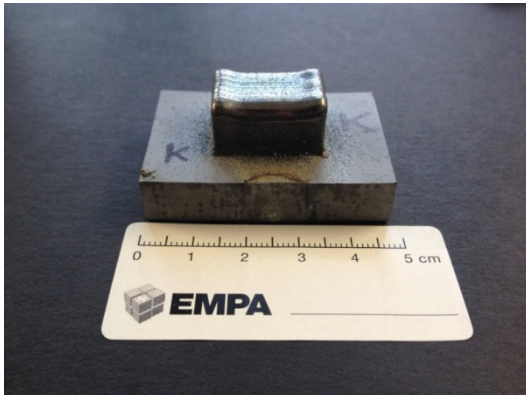
AM of TiAl with more complex geometries



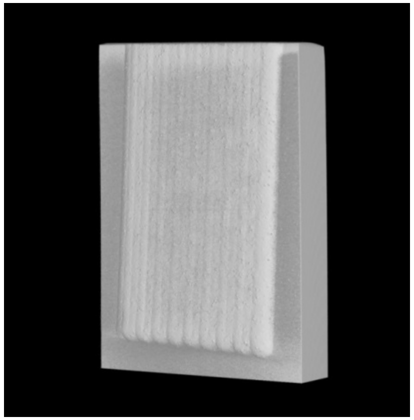


SLM 3D test structures (in collaboration with Inspire)





LMD test structure Ti-Al alloy (with TWI Ltd.)



CT of a LMD test specimen

note: structures were made from an Y_2O_3-ODS-variant

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New materials by AM

- Advantages of selective laser or electron beam melting
  - Short matter-beam interaction time due to high scanning speed
  - Small meltpool
  - High heating and cooling rates

⇨ Very fast material consolidation

⇨ Potential for processing of metastable materials, novel types of metal-matrix composites, or multi-material structures

  - Metal-diamond/metal-cBN composites
  - Novel High-entropy alloy components
  - …

New materials by AM - metal-diamond composites

(with A.B. Spierings, K. Wegener – Inspire/ETHZ)

- Metal-diamond composites interesting for high-performance cutting or grinding tools
- Conventional production: Galvanic Ni-bonding of diamond particles
  - Only single layer diamond tools possible, typically with simple geometry
- AM offers possibility to produce complexely shape geometries (e.g. internal cooling chanels
- Problem: Graphitization tendency of diamond particles at elevated temperatures
  - Depending on atmosphere (Inert atmosphere / vacuum ≈ 1’500° , Air ≈ 1’000°C)

Approach: *Brazing alloy as matrix material*

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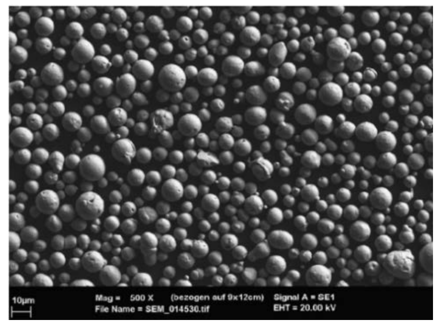
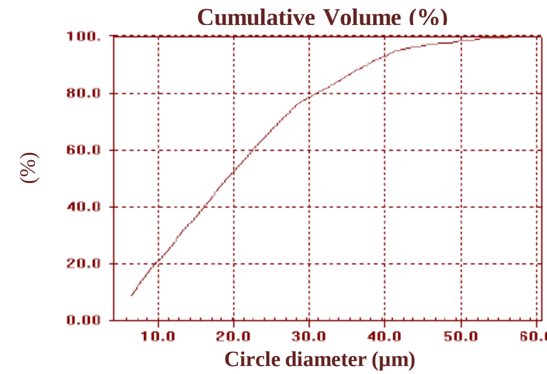
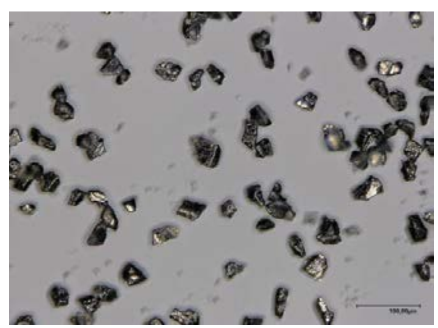
- **Matrix material**
  - Cu-based active brazing alloy
  - High thermal conductivity (> ≈ 55 W/mK)
  - $T_{liquidus}$ = 925°C
  - Powder with
    - D_{10} = 7.6µm
    - D_{50} = 20µm
    - D_{90} = 38µm

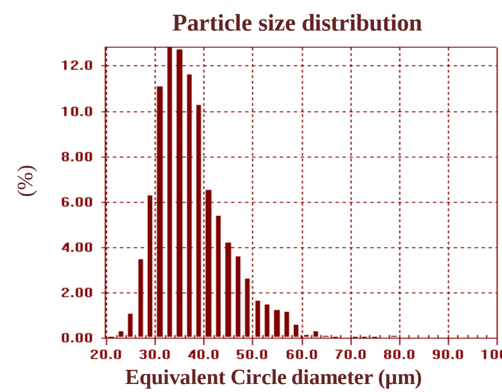
Composition	Cu	Sn	Ti	Zr
wt. %	73.9	14.4	10.2	1.5





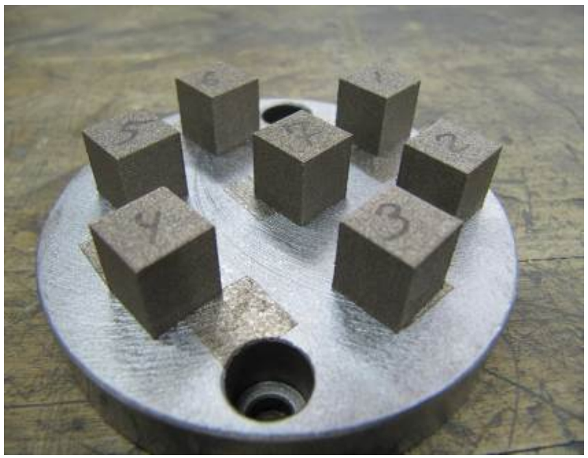
- **Diamond particles**
  - 50 vol% Ni-coated to protect the diamond particles from graphitization (additional heat sink)
  - Particles
    - Mean particle ∅ 33.9 ± 6.4µm

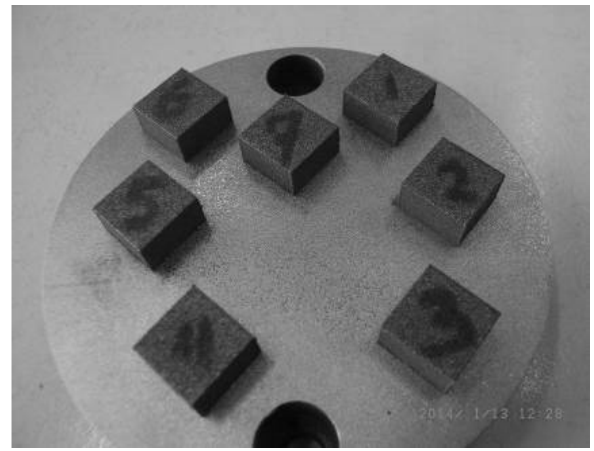




Metal-diamond composites

- SLM processing of a of brazing alloy & diamond particles
  - Stable specimens with good surface quality can be produced





SLM-samples of Diabraze with 10 vol% diamond (left) and 20 vol% Ni-coated diamond (right)

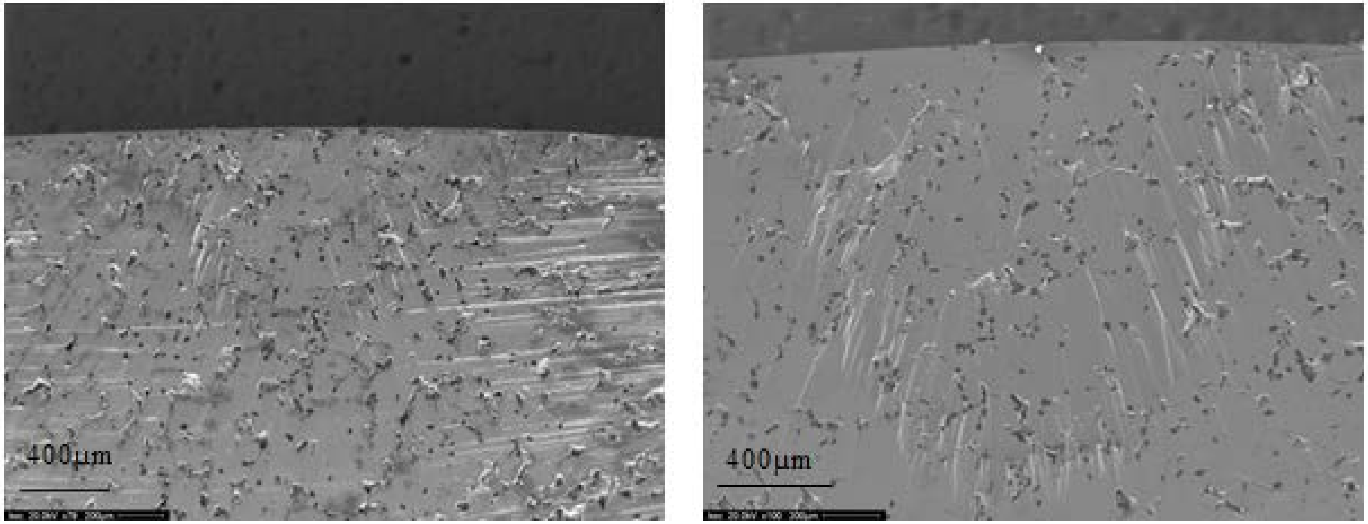
  - They are difficult to remove from the base plate.

  ... and have already a very good abrasive effect !

Metal-diamond composites

- SLM processing of a of brazing alloy & diamond particles

Ion cross-section milled SEM pictures of samples with 10 vol-% diamond particles



SEM (SE) - Micrographs of SLM-samples of Diabraze with 10 vol% Ni-coated diamond, energy input EL = 40.4 $^{J}/_{mm^3}$ (left) and EL = 50.5 $^{J}/_{mm^3}$ (right).

- Diamonds are homogeneously distributed in the matrix
- Diamonds survived almost unchanged
- Some remaining porosity and cracks are visible

Metal-diamond composites

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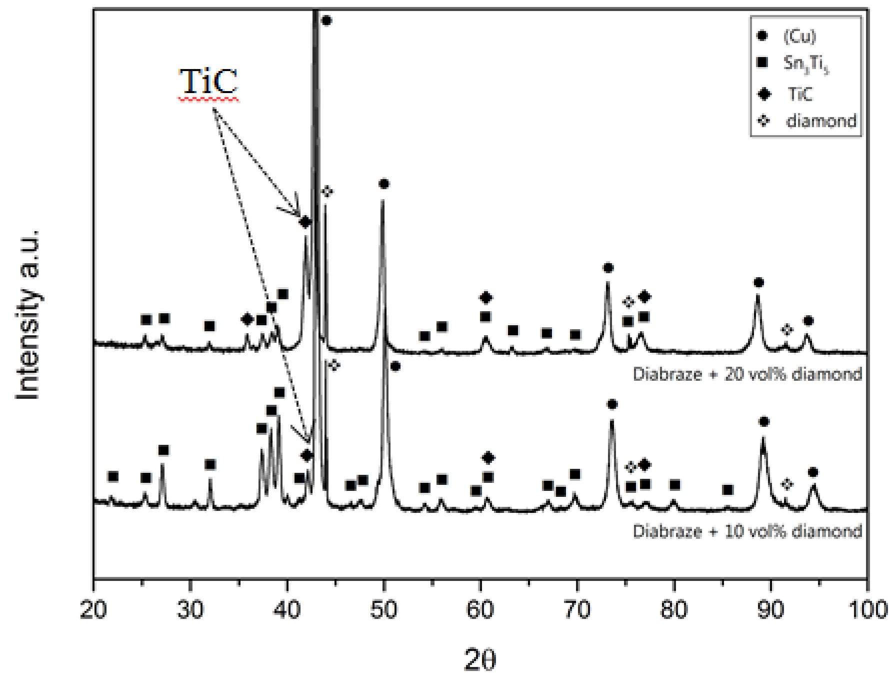
XRD spectra

- The matrix consists of a mixture of a Cu solid solution and $(Cu,Sn)_3Ti_5$ with Cu as the main phase.
- Formation of TiC is more pronounced for the 20 vol-% samples than for the 10 vol-% samples
- Ni fully dissolves in the matrix (Ni peak below treshold-level of XRD).
  ➔ Ni was detected by EDX.



XRD-spectra of Diabraze with 10 vol% Diamond (bottom) and 20 vol% Diamond (top). The identified phases are indicated.

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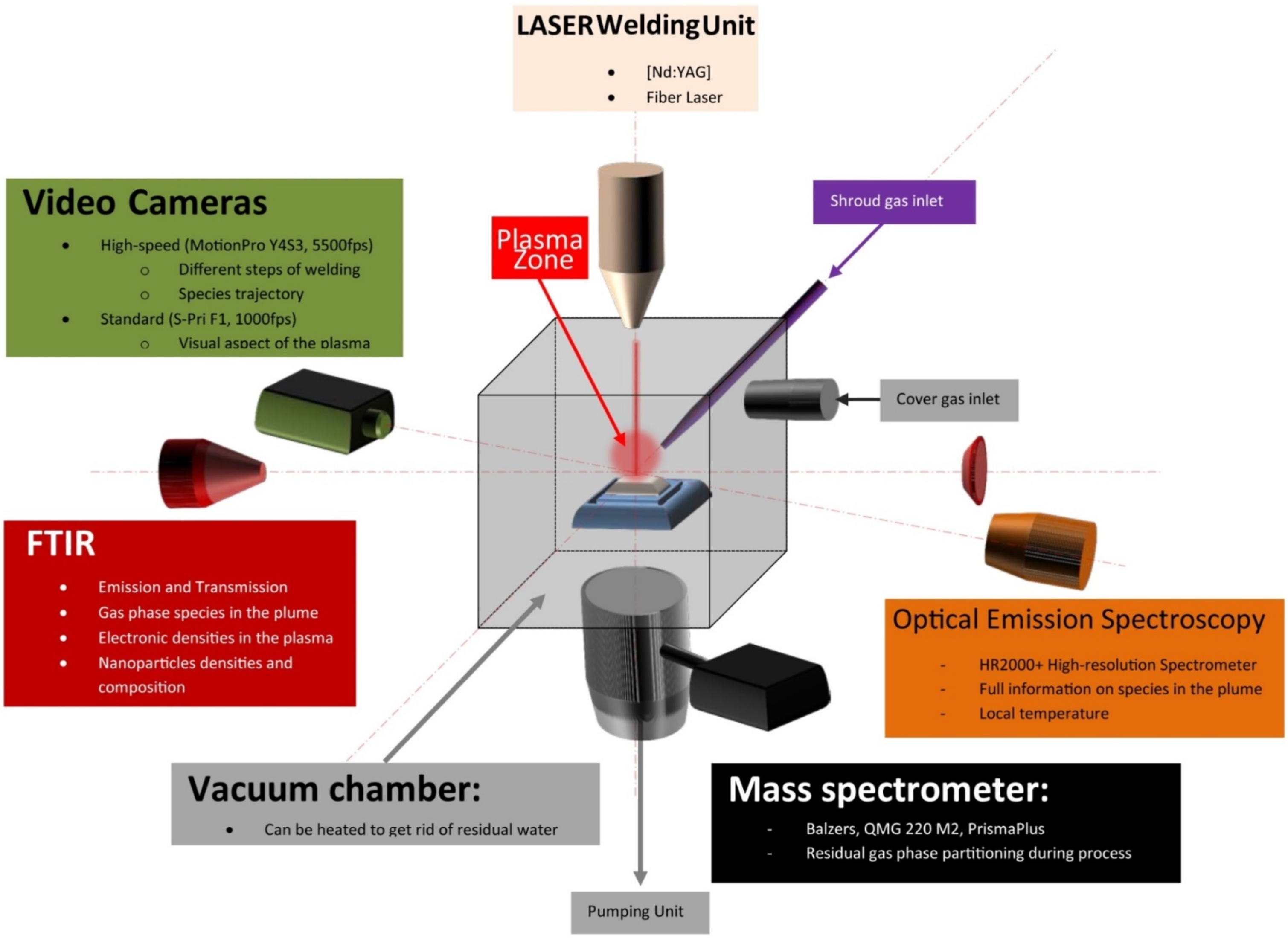
Visions

- In theory, AM allows for the fabrication of multi-material components and build-ups with complex geometries and new functionalities
  - Materials with integrated sensing capabilities (e.g. optical fibre gratings)
  - Graded structures with altered mechanical and physical properties
  - Repair of complexely shaped strcutures from composite materials
  - ...
- Besides a thorough understanding of the materials, this requires a new level of process control
  - Local variation of laser power, scanning speed...
  - Pre-/post-heating to adjust cooling rates
  - ...
- A control of the process requires a reliable online monitoring of the process
  - Melt pool geometries
  - Local temperatures
  - Effective laser power
  - ...

Set-up for in-situ monitoring of laser processing



The dream

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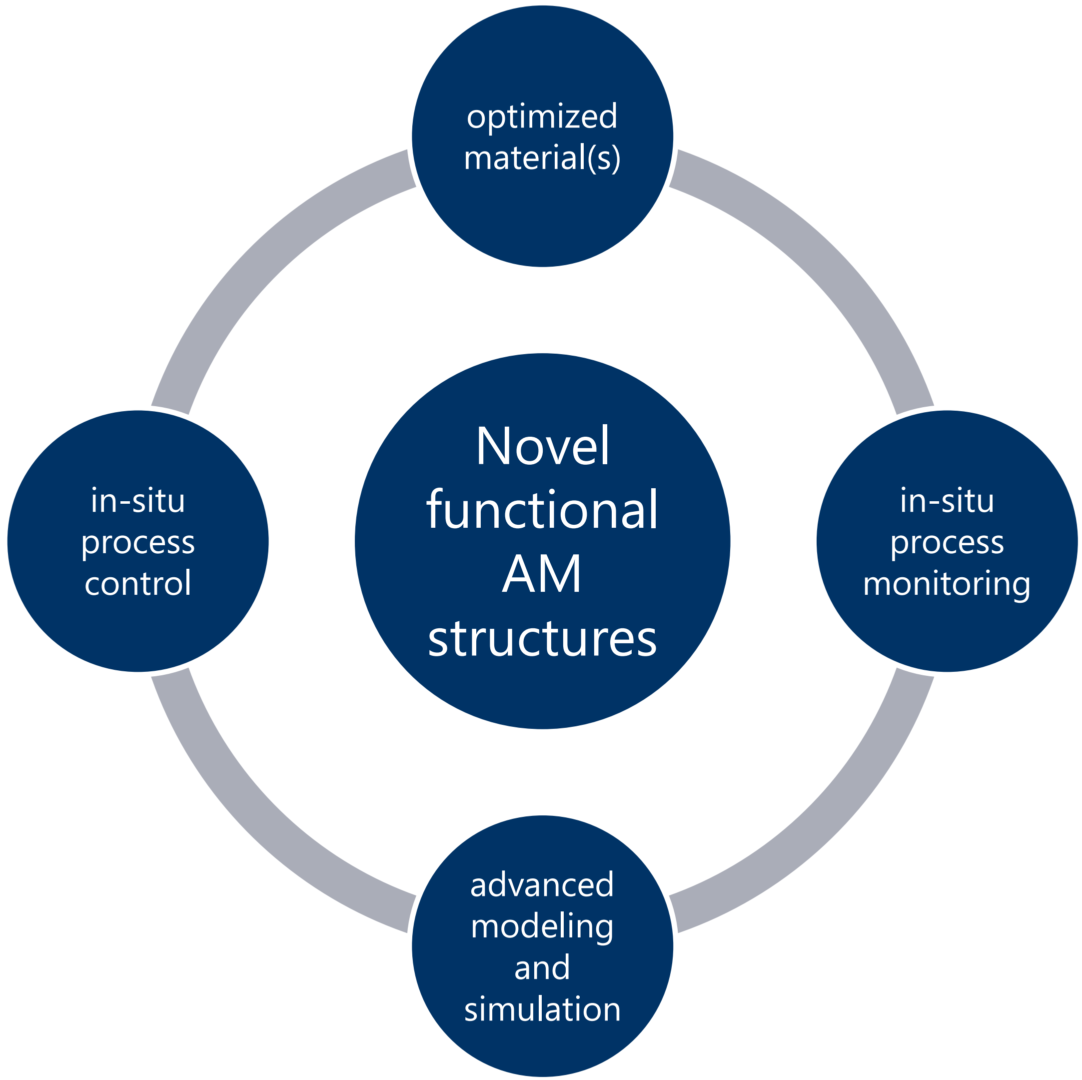

Thank you

Dr. Christian Leinenbach
Head Alloy Design and Processing Technologies
Laboratory for Joining Technologies and Corrosion
Empa
Swiss Federal Laboratories for Materials Science and Technology
Überlandstrasse 129
8600 Zurich/Dubendorf
Switzerland
Tel +41 58 765 4518
Fax +41 58 765 1122
christian.leinenbach@empa.ch
www.empa.ch